No 3

Chloride of methyl as antiseptic. 1886

Use steep H2O in mashing

A.D. 1886, 8th FEBRUARY. N° 1820.

PROVISIONAL SPECIFICATION.

A New or Improved Diastasic Saccharine Substance, and Method of Manufacturing the same.

I, LEON CUISINIER, of Brussels in the Kingdom of Belgium, Chemist do hereby declare the nature of this invention to be as follows:—

No solid diastasic saccharine substance has been hitherto discovered, except the solid maltose discovered by Dubrunfaut.

I have, however, discovered a fresh substance of a much more sugary flavour, and capable of being more easily manufactured than the solid maltose, to this body, I have given the distinctive name of "cerealose."

Cerealose is produced by a series of reactions difficult to explain in view of the uncertainty of our present chemical knowledge with regard to amylaceous bodies. I will therefore simply describe what I have discovered, and reserve myself the option of setting forth in subsequent patents of addition the explanation of the facts herein set forth.

I. If maize be simply steeped in cold water for two or three days, a special kind of diastase is developed, both in the body of the steeped grains, and in the water used. This diastase I call "glucase" and its characteristic reaction is the conversion of starch directly into a saccharine substance different from maltose, and which appears according to preliminary analysis to be dextrose or grape sugar. This glucase is obtainable not only from steeped maize grains, and the steeping water of the same but also from those of any cereal treated in the same manner.

II. Glucase transforms starch into dextrose it is almost entirely without the liquefying action which sprouted barley has upon starch paste, it has but little action upon starch and upon dextrine and apparently none at all on maltose. The aid of a reagent like fresh malt having a strong liquefying action on starch accelerates the action of the glucase; these two diastases therefore appear to mutually assist each other's reaction. The preliminary heating of the charge to the highest temperature to which it can be raised without affecting the starch granules, also facilitates the action of the glucase. In charges of maize this limit of temperature is about 67 degrees centigrade, the action of the glucase is destroyed by heating to between 67 degrees and 70 degrees centigrade, but it is still very energetic between 60 degrees and 65 degrees.

The process I prefer to employ is based upon the above considerations.

Accordingly rice or maize for instance is steeped in cold water for two or three days, after which the steeping water, which is usually very slightly acid is drawn

[*Price* 6*d*.]

off and preserved for future use. The steeped grain is crushed between several successive pairs of steel cylinders. The meal thus obtained is immediately mixed with twice its weight of warm water at 67 degrees centigrade. There is also added from two and a half per cent to five per cent of green crushed malt, per 100 kilos of steeped and ground grain, and the whole is then heated by steam in a double bottom mixing pan, so as to maintain the temperature at about 67 degrees for two or three minutes. The charge is then cooled by pouring the steeping water into it, but the temperature must not be reduced below 60 degrees or 62 degrees. The charge is then placed by itself in a mash tub provided with an agitator by means of which the charge is stirred from time to time. The temperature must be strictly maintained between 60 degrees and 62 degrees throughout the whole period of maceration, say forty eight hours or more by a steam jacket on the agitator for instance. A charge prepared of the density of 20 or 25 kilos of material per hectolitre can be preserved without accident during the maceration. It is in any case easy to prevent any alteration by the use of chloroform or of chloride of methyl, chloride of methyl especially is easily made and used, it may be prepared by heating in a lead lined alembic a mixture of methylic alcohol sulphuric acid and sea salt. The gas evolved is washed and collected in a gasometer from which it can be drawn by a pump, and forced into the agitators, so as to charge the solution with about a three thousandth part. When the maceration is finished, it is only necessary in order to set the chloride of methyl free to create a slight vacuum above the liquid by means of a special air pump which passes the gas into vessels containing absorbing substances such as alcohol or crystallizable acetic acid. The maceration is completed when the density of the liquid ceases to increase. The residues are then separated, preferably by means of filter presses, on account of the fluidity of the liquid. The residues still retaining a little starch which can only be rendered soluble by malt by steaming under pressure, they may be utilized for making maltose syrup for breweries by the well known processes. By the above described process, at least three quarters of the extractable matter contained in the grain is obtainable. The filtered juice is clear, and does not show much turbidity on concentration, nevertheless in order to obtain a perfectly clear liquor, a second filtration is indispensable.

When the juice is concentrated to a syrup of 40 degrees, it is run into moulds and capped, either with a quantity of solid glucose of commerce, or with a little cerealose remaining from a previous operation; a vigorous conglomeration ensues in the course of a few hours, 24 at the outside, and the very consistent lumps can then be removed from the moulds.

Dated this 6th day of February 1886.

WM P. THOMPSON & Co.,
Of 6, Lord Street, Liverpool,
Patent Agents for Applicant.

COMPLETE SPECIFICATION.

A New or Improved Diastasic Saccharine Substance, and Method of Manufacturing the same.

I, LÉON CUISINIER, of Brussels in the Kingdom of Belgium, Chemist, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

No solid diastasic saccharine substance has been hitherto discovered except the solid maltose discovered by Dubrunfaut.

I have however, discovered a fresh substance of a much more sugary flavour and capable of being more easily manufactured than the solid maltose, to this body I have given the distinctive name of "cerealose."

Cerealose is produced by a series of reactions difficult to explain in view of the uncertainty of our present chemical knowledge with regard to amylaceous bodies. I will therefore simply describe what I have discovered, and reserve myself the option of setting forth in subsequent patents of addition the explanation of the facts herein set forth.

1. If maize be simply steeped in cold water for two or three days, a special kind of diastase is developed, both in the body of the steeped grains, and in the water used. This diastase I call "Glucase" and its characteristic reaction is the conversion of starch directly into a saccharine substance different from maltose, and which appears according to preliminary analysis to be dextrose or grape sugar. This glucase is attainable not only from steeped maize grains and the steeping water of the same, but also from those of any cereal treated in the same manner.

2. Glucase transforms starch into dextrose it is almost entirely without the liquefying action which sprouted barley has upon starch paste, it has but little action upon starch and upon dextrine and apparently none at all on maltose. The aid of a reagent like fresh malt having a strong liquefying action on starch accelerates the action of the glucase; these two diastases therefore appear to mutually assist each other's reaction. The preliminary heating of the charge to the highest temperature to which it can be raised without affecting the starch granules also facilitates the action of the glucase. In charges of maize this limit of temperature is about 67 degrees centigrade, the action of the glucase is destroyed by heating to between 67 degrees and 70 degrees centigrade, but it is still very energetic between 60 degrees and 65 degrees.

The process I prefer to employ is based upon the above considerations.

Accordingly, rice or maize for instance is steeped in cold water for two or three days, after which the steeping water, which is usually very slightly acid is drawn off and preserved for future use. The steeped grain is crushed between several successive pairs of steel cylinders. The meal thus obtained is immediately mixed with twice its weight of warm water at 67 degrees centigrade. There is also added from two and half per cent to five per cent of green crushed malt per 100 kilos of steeped and ground grain, and the whole is then heated by steam in a double bottom mixing pan, so as to maintain the temperature at about 67 degrees for two

or three minutes. The charge is then cooled by pouring the steeping water into it, but the temperature must not be reduced below 60 degrees or 62 degrees. The charge is then placed by itself in a mash tub provided with an agitator by means of which the charge is stirred from time to time. The temperature must be strictly maintained between 60 degrees and 62 degrees throughout the whole period of maceration, say forty eight hours or more by a steam jacket on the agitator for instance a charge prepared of the density of 20 or 25 kilos of material per hectolitre can be preserved without accident during the maceration. It is in any case easy to prevent any alteration by the use of chloroform or of chloride of methyl, chloride of methyl especially is easily made and used, it may be prepared, by heating in a lead lined alembic a mixture of methylic alcohol, sulphuric acid and sea salt. The gas evolved is washed and collected in a gasometer from which it can be drawn by a pump and forced into the agitators so as to charge the solution with about a three thousandth part. When the maceration is finished it is only necessary in order to set the chloride of methyl free, to create a slight vacuum above the liquid by means of a special air pump which passes the gas into vessels containing absorbing substances such as alcohol or crystallisable acetic acid. The maceration is completed when the density of the liquid ceases to increase. The residues are then separated, preferably by means of filter presses, on account of the fluidity of the liquid. The residues still retaining a little starch, which can only be rendered soluble by malt by steaming under pressure. They may be utilized for making maltose syrup for breweries by the well known processes. By the above described process, at least three quarters of the extractible matter contained in the grain is obtainable. The filtered juice is clear, and does not show much turbidity on concentration, nevertheless in order to obtain a perfectly clear liquor, a second filtration is indispensable.

When the juice is concentrated to a syrup of 40 degrees it is run into moulds and capped either with a quantity of solid glucase of commerce, or with a little cerealose remaining from a previous operation a vigorous conglomeration ensues in the course of a few hours, 24 at the outside, and the very consistent lumps can then be removed from the moulds.

Cerealose possesses the following composition which I state in comparison with that of glucase and of maltose.

	Cerealose.			
	Pure Glucase.	No. 1 Sample.	No. 2 Sample.	Maltose.
Dextrose - - - -	100	72·97	48·00	—
Maltose - - - -	—	5·94	24·00	100
Dextrine - - - -	—	1·18	12·80	—
R = rotation of the solution at 100 qr per litre - -	48	45·5	76·8	126
G = reducing power of the same solution calculated as dextrose -	100	76·0	62·4	60
Proportion $\frac{R}{G}$ - - -	48	55	123	210

The proportion $\frac{R}{G}$ which I usually make use of in my investigations clearly shews that this saccharine body is perfectly distinct from commercial glucase and maltose. Cerealose contains a notable quantity of the salts phosphoric acid and soluble nitrogen of the grain it is made from. It is absolutely free from sulphates and from lime salts, as well as from other deleterious substances due to the use of impure acids, this at once distinguishes it from the solid glucase of commerce. Its

composition is comparable to that of the syrups of maltose used in brewing beer; it ferments rapidly and gives a yield of 44 litres of 100 per cent alcohol per 100 kilos of dry substance.

Having now particularly described and ascertained the nature of the said invention, and in what manner the same is to be performed, I declare that what I claim is:—

1. The exclusive right of manufacturing the new saccharine substance described and called "cerealose" the properties composition, and method of manufacture of which are set forth above.

2. The use of the reactions of the new diastase discovered by me and named "glucase" together with the liquefying properties of malt for the purpose of inducing a glucase saccharification.

3. The use of starchy bodies for the production of cerealose.

4. The combination of processes hereinbefore described for the manufacture of cerealose.

5. The use of chloride of methyl to prevent alteration of the materials under operation.

6. The application of cerealose to the production of beer and of alcohol, for sugaring wines, for sweetening confectionery, chocolate, bonbons, and the like and similar purposes.

Dated this 22nd day of September 1886.

WM P. THOMPSON & Co.,
Filing Agents,
323, High Holborn, London.

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